PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Compositions and methods for producing electrically conductive coatings.

We, ACHESON INDUSTRIES, INC., a Corporation organised and existing under the laws of the State of Michigan, United States of America, of 321 Michigan National Bank Building, Port Huron, Michigan, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to compositions producing electrically conductive coatings on supporting surfaces.

In our Application No. 26285/66 we describe and claim a composition for use in forming electrically conductive coatings which comprises, in weight percent, 10% to 25% of a conductive pigment, 15% to 80% of a solution of an alkali metal silicate containing from 42% to 71% water, 0.3% to 5% of a water-soluble or water-dispersible organic resin material decomposible at the temperatures normally employed in cathode ray tube manufacture, i.e. 250 to 475°C., 0.5% to 10% of an alkali metal or ammonium carboxylic acid salt and the balance water. This composition may be used for forming an electrically conductive coating on a surface, for example by applying the composition thereto and heat curing the composition on the surface at a temperature within the range of 250 to 475° until the coating is hard and adherent to the surface.

The compositions have particular utility in coating the surface of cathode ray tubes. For this purpose the coating is preferably 0.1 to 2 mils. Such compositions have improved adhesion, improved hardness, improved resistance to abrasion, improved outgassing properties when the tubes are evacuated and a decreased tendency for disintegration of the coating during use.

It has now been found that smaller quantities of carboxylic acid salt than 0.5% can be used successfully in such compositions. It has been found that the reduced organic content improves the outgassing properties of the coated vacuum tubes as there is less undesirable material to remove, when the amounts of the other components of the composition are kept the same. Amounts as low as 0.15%, or even 0.05% by weight are satisfactory. It has also been found that the low salt level does not affect adversely the poroxity promoting tendency or the coating properties, thus reducing the cost of the composition by decreasing the manufacturing controls necessary to maintain the salt content within 0.5 to 10%. Accordingly, the present invention provides a composition for use in forming electrically conductive coatings, which comprises, in weight percent, 10% to 25% of a conductive pigment, 15% to 80% alkali metal silicate containing from 42% to 71% water, 0.3% to 5% of a water-soluble or dispersible organic resin material, 0.05% to 0.5% of an alkali metal or ammonium carboxylic acid salt and the balance water. The compositions may also comprise a dispersing agent to obtain proper rheological properties. Suitable such dispersing agents include sodium lignin sulphonate and naphthalene sulphonic acid condensates.

A preferred composition of this invention has the following composition:

Conductive Pigment	*Weight Percent*
Graphite, carbon black, finely divided metal, or mixtures	12—20
Binder (alkali metal silicate — 42%—71% water)	35—75
Organic resinous materials (water soluble or dispersible resins)	0.75—2.0
Alkali metal carboxylic acid salt	0.15—0.5
Water (Distilled or Deionized)	Balance

Preferably the alkali metal silicate is potassium silicate, the preferred resin is polyvinylpyrrolidone and the preferred alkali metal carboxylic acid salt is potassium sodium tartrate.

[Price 5s. 0d.]

The compositions of this invention may be prepared simply by blending the selected quantity of each of the components and mixing until a uniform dispersion is obtained. No particular order of addition of the components is necessary and any of a variety of standard mixing equipment may be used to form the dispersions.

Further features of the compositions of this invention are described in greater detail in our Application No. 26285/66, to which reference should be made.

The present invention also comprises a method for forming an electrically conductive coating on a surface which comprises applying to the surface to be coated for example by spraying, brushing, flowing, or roller-coating a composition of the present invention so as to deposit on the substrate the desired quantity of that composition. Preferably, for cathode ray tube coatings, a sufficient quantity of the composition is applied to produce a cured coating thickness of 0.1 to 2 mils. Thereafter, the coating may first be dried with circulating hot air or the like, and then heat-cured at a temperature of from 250°C to 475°C., for example for one-half to two and one-half hours until the coating is hard and adherent to the surface. Alternatively, the preliminary hot air drying step may be omitted and the coated surface slowly raised from room to its final curing temperature. After curing, the coating may, if desired, be rinsed with water, or it may be used in its heat cured condition without further treatment.

The following Example further illustrates this invention.

EXAMPLE

A coating composition was prepared from the following materials, in weight percent:

	Weight Percent
Graphite (maximum particle size — 10 microns)	14.2
Polyvinylpyrrolidone (average molecular weight—10,000)	1.5
Aqueous potassium silicate (30° Baume at 68°F, viscosity — 7 centipoises at 68°) 9.05% K_2O and 19.9% SiO_2)	74.1
Deionized water	10.2

The above ingredients were mixed thoroughly for 16 hours in a standard pebble mill. The dispersion removed from the mill was applied to a 3" x 6" glass panel, at room temperature, by spraying. The coated panel was cured in an air atmosphere furnace slowly raised to a temperature of 400°C with the panel being maintained at 400°C. for approximately one hour.

When the panel was removed from the furnace and inspected under a microscope at magnifications up to 60 diameters the coating was observed to be smooth, glassy and free of pores. The coating was measured for electrical resistance and found to have a resistance of 126 ohms per square. The adhesion and hardness of the film was tested by scraping a blunt metal blade across the coating. The glassy skin of the film was easily pierced and portions of the coating removed in the form of flakes.

A similar composition was then prepared, which contained, in addition, Rochelle salt and sodium lignin sulphonate. It had the following composition:

	Weight Percent
Graphite (maximum particle size-10-microns)	14
Aqueous potassium silicate (29% solids)	29
Polyvinylpyrrolidone (average molecular weight, 40,000)	0.2
Rochelle Salt (potassium-sodium tartrate)	0.2
Sodium Lignin Sulphonate (dispersing agent)	0.4
Deionized water balance	56.2

A glass panel was sprayed with this composition and then heat-cured under identical conditions to those employed for the first composition. Visual inspection of the panel, without magnification, showed that the surface was a porous matt coating. Microscopic examination revealed that the pores were distributed over the entire surface.

Using the same adhesion test as described above the film was found to be substantially harder and more adherent than the other coating. No penetration or flaking of the coating was obtained in the test.

WHAT WE CLAIM IS:—

1. A composition for use in forming electrically conductive coatings which comprises, in weight percent, 10% to 25% of a conductive pigment, 15% to 80% alkali metal silicate containing from 42% to 71% water, 0.3% to 5% of a water-soluble or dispersible organic resin material, 0.05% to 0.5% of an alkali metal or ammonium carboxylic acid salt and the balance water.

2. A composition according to claim 1 which comprises, in weight percent, 12% to 20% of a conductive pigment, 35% to 75% alkali metal silicate containing from 42% to 71% water, 0.75% to 2% of an organic resin as defined in claim 1, 0.15% to 0.5% of an alkali metal or ammonium carboxylic acid salt and the balance water.

3. A composition according to claim 1 or 2 wherein the alkali metal silicate is potassium silicate.

4. A composition according to any one of claims 1 to 3 wherein the resin is polyvinylpyrrolidone.

5. A composition according to any one of the preceding claims which comprises 0.15% to 0.5% of an alkali metal or ammonium carboxylic acid salt.

6. A composition according to any one of the preceding claims wherein said alkali metal carboxylic acid salt is potassium sodium tartrate.

7. A composition according to any one of the preceding claims which also comprises sodium lignin sulphonate or a naphthalene sulphonic acid condensate.

8. A composition according to claim 1 substantially as hereinbefore described.

9. A composition according to claim 1 substantially as described in the Example.

10. A method for forming an electrically conductive coating on a surface which comprises applying to the surface a composition as claimed in any one of the preceding claims and heat curing the composition on the surface at a temperature of from 250°C. to 475°C until the coating is hard and adherent to the surface.

11. A method according to claim 10 wherein the said surface is a surface of a cathode ray tube and the amount of the composition is such that the coating has a thickness of 0.1 to 2 mils.

12. A method according to claim 10 substantially as hereinbefore described.

13. An article having on a surface thereof an electrically conductive coating formed by a method as claimed in any one of claims 10 to 12.

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